Pollution Prevention Technology Application Analysis

Utilizing

"PICKLEX®

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Prepared using the EPA Pollution Prevention Technology Application Analysis Template

U.S. Environmental Protection Agency - New England 1 Congress Street, Suite – 1100 (SPN) Boston, MA 02114-2023 (617) 918-1111

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DISCLAIMER

This document describes "Picklex®, a metal surface preparation and pre-treatment technology for use prior to metal finishing. EPA's Pollution Prevention Technology Application Analysis Template was used by International Chemical Products, Inc., the vendor of Picklex®, as a format for helping the reader understand the technology. While the Template format covers many general questions that you should ask when evaluating a P2 technology, it may not include all the questions that are relevant to your business, or which your business is legally required to ask.

This document is not an official U.S. EPA guidance document and should not be relied upon as a method to identify or comply with local, state, or federal laws and regulations. EPA does not endorse any technology analyzed herein.

Introduction

The purpose of this technology application analysis is to summarize the results of utilizing new and innovative pollution prevention technology, and promote the use of P2 new technology, which has been utilized in a full scale commercial application. In this case, the new technology is an alternative process for metal surface preparation and pre-treatment, before any metal finishing, with a unique environmentally safe (negligible VOC), non-hazardous, non-toxic, non-flammable, and water soluble chemical product called "Picklex®".

Picklex® has **Zero Waste Discharge**, eliminating a major part of pollution in the finishing industries. The application of this new technology & product is to assist vendors of Pollution Prevention Technologies; either to apply this process and product to their needs or develop their own technology application analysis using the product.

Picklex® can be used in surface preparation and pre-treatment for any metal finishing system, such as electroplating, anodizing, painting, e-coating, powder coating etc. The technology application analysis presents an application of this new alternative process, being used in a liquid painting and powder coating plant in Texas. Also, demonstrate the degree of pollution prevention achieved, along with the considerable reduction in process time and cost. In addition, the quality of finishing is much superior than the finishing presently done by traditional methods.

NOTE: This alternate process with Picklex® is presently in process of evaluation by **US EPA**, as a possible replacement of hazardous/toxic chemicals from surface preparation/pre-treatment process (letter attached).

This template serves as guidance for characterizing in a concise manner, the main features of the technology and the product, the costs associated with implementation, regulatory aspects, and lessons learned from the application experience.

The technology application analysis illustrates how the P2 template can summarize a technology and the intent of the EPA in developing the P2 template is to promote the use of technology application analysis as a method of promoting and accelerating the introduction and use of new P2 technologies.

This application analysis is divided into seven sections:

- Introduction
- Description of P2 Technology
- P2 Technology Application
- P2 Technology Performance
- Cost Information
- Regulatory / Safety Requirements
- Lessons Learned / Implementation Issues

Description of P2 Technology

In any metal finishing process, the metal surface preparation and pre-treatment plays a big role towards the quality of the final finish. If the metal surface is not cleaned properly or if the surface has contamination such as oil, rust etc., the adhesion between the metal and the top coat becomes poor, resulting in a premature failure of the top coating. Also, a pre-treatment is needed to provide a protective coating for corrosion resistance. The degree of cleanliness often depends on the final quality requirements of the finishing job. The quality test requirements of a finishing can be generalized into some of these following tests:

- Adhesion test
- Bending test
- · Gloss or texture of the surface
- Salt spray test for certain number of hours
- Chemical resistance test
- Impact test

To obtain a reasonably clean surface, the metal parts must undergo a series of hazardous and/or toxic chemicals, such as acid wash (to remove rust and etch the surface), phosphate treatment (to give some corrosion protection) etc. All of these chemicals are usually heated to fairly high temperature (around 160 - 180° F), creating a hazardous working condition with the chemical fumes. Also, from time to time, these chemicals along with the rinse water, has to be disposed, which according to EPA, are categorized as hazardous waste. These hazardous wastes from all kinds of metal finishing places, adds up to be a very large amount of pollution.

The following section describes an alternate process for surface preparation and pre-treatment of metal, before any metal finishing, which is performed by **Picklex®**. **Picklex®** works at room temperature, with no waste and provides an equivalent to a perfectly clean surface, ready for finishing. This process eliminates all the hazardous/toxic chemicals, such as acid, phosphate etc. Also, the process requires less number of steps than the traditional process for surface preparation/pre-treatment. The applicability of this technology and the product to different types of finishing industries are also described. In addition, the advantages and the benefits of this technology/product are provided.



Overview of the finishing processes

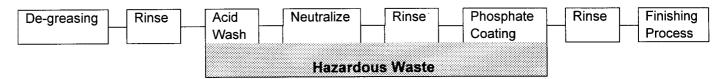
The surface preparation/pre-treatment for Metal Finishing systems and the Pickling Process are described below.

1) Metal finishing Process

Comparison between traditional process and Picklex® Process (Patent pending)

Traditional Surface Preparation/Pre-treatment Process:

A typical traditional surface preparation and pre-treatment process for any metal finishing system can be summarized as follows (a seven stage process):



NOTE: The above process has Hazardous Waste Disposal from Acid through Phosphate treatment

The finishing process shown at the end, is the beginning of the final finishing after surface preparation and pretreatment of metal parts, which in case of powder coating, starts with oven heating, and then to powder coating and curing. For electroplating, anodizing, e-coating and liquid painting, the metal part goes to the finishing solutions (e.g., electroplating solution after acid etching, anodizing solution, e-coat paint or liquid paint, etc.).

The process with Picklex® is a standard Four Step Process as shown below:



Standard Four Step Process

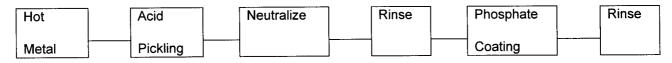
How Picklex® works: Picklex® cleans the surface by removing dust, light oil and surface rust, conditions the metal surface, chemically converts 100% rust/oxidation (including micro-rust) and reacts with the metal on the surface to form a conductive, metallurgical protective coating, all in one application. NOTE: Because the oil on the surface can be heavy oil or grease, which Picklex® may not be able to remove, therefore it is recommended to have the de-greasing done separately, which will remove oil and any soil from the surface.

Zero Waste from Picklex®: Picklex® does not lose concentration when used. It will work continuously, if the rust particles are properly filtered and the chemical is kept reasonably clean. In case the steel surface has negligible rust or in case of aluminum, the filtering system may not be needed (this has to be evaluated according to the condition of the metal).

This above process eliminates three steps from a typical surface preparation/pre-treatment process, along with a considerable reduction of waste disposal, process time and completely eliminates the hazardous waste disposal.

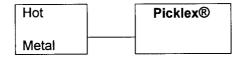
2) Pickling Process

A typical metal pickling process (de-scaling/pickling) after heat treatment (annealing) is shown below:



NOTE: The above process has Hazardous Waste Disposal from Acid, Phosphate and rinse water.

The One Step Pickling/De-scaling/Coating Process ** by Picklex® is shown below :



** Patent pending

One Step Process

How Picklex® works in Pickling: After heat treatment, the metal temperature is brought down to around 150° F-200° F and dipped in Picklex® at room temperature. In one dipping process, Picklex® de-scales, conditions the metal and provides a metallurgical protective coating, making the surface with zero rust/scale, ready for painting and welding. This process replaces the complete traditional process of acid pickling.

Zero Waste Disposal : The scale particles are filtered and the chemical is re-cycled in the tank. Since **Picklex®** does not lose concentration when used, the process is continuous, when properly filtered.



This section describes the applicability of this technology to users, the development history, and the advantages/limitations claimed by the technology vendor.

Applicability to industry/user

The primary focus of this P2 technology by the unique product **Picklex®**, has been directed towards the following metal finishing industries :-

- Electroplating
- Anodizing
- Powder coating
- E-coating
- Liquid painting

Also, it is directed to the manufacturing industries dealing with heat treatment of metal (annealing process), who is involved with **acid pickling process**.

It is due to following reasons:

- Hazardous/Toxic Waste created by the chemicals used in traditional metal surface preparation and pretreatment process
- Hazardous/Toxic fumes created by the chemicals due to heating of the chemicals in the surface preparation process
- Rinse water containing large amount of metal from acid etching
- Great deal of expense, time and labor involved in disposing or recovering hazardous waste
- Extreme health hazard at working place

The process with **Picklex**® eliminates hazardous waste, and because it works at room temperature, there are no strong chemical fumes.

In addition to eliminating waste, this process results in much better quality finishing in any finishing process, than the conventional methods.

Picklex® is an environmentally safe, non-toxic, non-hazardous, non-flammable, and water soluble product. **Picklex®** does not fall under any EPA, OSHA or D. O. T. regulation.

PICKLEX® simultaneously combines the following processes, when a metal (hot or cold rolled steel, and also aluminum) is treated for a certain length of time (usually less than the traditional methods).

Cleans the surface by removing light mill oil and dust

- · Removes surface rust/oxidation and mill scale
- Converts 100% of the micro-rust to a protective coating
- · Acts as a Rust inhibitor and stops Creeping or spreading rust
- Provides a Conductive Metallurgical Protective coating for top coating purposes.
- Metal surface conditioner
- Prepares a zero rust/oxidation surface ready for any metal finishing.
- Increases Weld strength considerably, when welding is performed on the coated surface

Development / Application History

Table 1 – Application History of Picklex® Technology

Time	Location	Scale	Target Metal	Purpose	Results
June 1997	Huntsville, AL	Lab Test	Steel	Painting	Passed 1540 hours of salt spray test with minimum surface preparation
Feb 1998	Huntsville, AL	Lab Test	Aluminum	Anodizing	Passed 500 hours of salt spray test with minimum surface preparation
June 1998	Hughes Parker Co., Lawrenceburg, TN	Field test	Steel	E-coat and Powder Coating	Passed all required quality tests. Also, passed 1500 hours of salt spray test in both cases (ASTM B117).
March 1998	Custom Finishing Co., AL	Field test	Aluminum	Powder Coating	Passed all quality tests with 500 hours of salt spray test
April 1998	Savage Precision Fab., Wylie, Tx	Commercial operation	Steel and Aluminum	Powder Coating & Liquid painting	Meeting all quality requirements, along with a reduction of around 50% on Process time and Running cost. Zero rejection for more than a year
Sept 1998	US EPA Cincinnati, OH	Evaluation & Testing	Steel	Electroplating	Passed all required quality tests
Nov 1998	US EPA Cincinnati, OH	Evaluation & Testing	Aluminum	Anodizing	Passed all required quality tests including 500 hours of salt spray test.

<u>Advantages</u>

The benefits and the advantages are shown on the next section, "P2 Technology Application" on page 7, where the actual benefits are being achieved by the host plant in Texas, using this alternate process with **Picklex®** in their commercial operation.

P2 Technology Application

This following section describes the use of new P2 technology, the alternate process for surface preparation and pre-treatment by using **Picklex®**, at Savage Precision Fab, Wylie, Texas, for their liquid painting and powder coating systems. This section will further describe the details of the P2 technology location within the plant and how the plant production / operation was affected.

P2 Technology Application

General Setting

Description of the fabricating/finishing plant :

Plant name : Savage Precision Fab Co.

Location : Wylie, TX

Nature of plant : Aircraft Parts fabricating and finishing (steel & aluminum),

commercial operation

Finishing Process : Liquid Painting & Powder Coating (Low volume)

Starting date with new process : Beginning of April, 1998

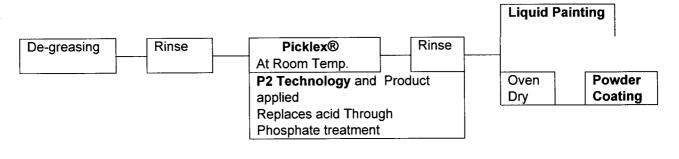
The above plant had a process for liquid painting system for a long period of time. Early this year they wanted to add a separate powder coating system, and also to minimize hazardous waste & pollution.

Process with Picklex®: The company replaced their old surface preparation/pre-treatment process for their liquid paint system, which used hazardous chemicals before, with four step alternate process with **Picklex®**. They also set up a new powder coating system, using the same surface preparation process.

P2 goals and objective: P2 goals and objective were to reduce hazardous waste and pollution on land and air. In old system, the hazardous waste from the chemicals has to be disposed from time to time. Also, the objective was to make the air cleaner inside from the chemical fumes and make the working conditions better.

Technology Implementation At Manufacturing / Industry Plant Site

This section will give a diagram showing where the P2 technology fits within the context of the host plant in Texas, where it has been applied.



In the above surface preparation and pre-treatment process, there is no hazardous waste.

The same surface preparation process is used for both liquid painting and powder coating (also, for both steel and aluminum).

The new P2 technology, which is the alternate process with **Picklex**®, replaces the old system with hazardous chemicals, such as acid, phosphate etc.

The P2 technology eliminated all hazardous waste, also minimized the overall waste in the surface preparation process considerably (with zero waste for **Picklex®**). Also, this specific P2 technology reduces the process time and running cost considerably, along with a higher quality surface preparation, which enhances the final finishing. The finishing has longer-term corrosion protection than before.

Benefits and advantages obtained by using the alternate process:

- Eliminated hazardous waste disposal problem (Zero Waste achieved)
- Eliminated acid and phosphate
- Eliminated most of the chemical fumes, health hazard problems
- Better working conditions by eliminating hazardous fumes
- ♦ Use less space due to less number of steps (four steps only)
- ◆ No pump or filtering system was necessary for the **Picklex®** tank, because the parts are fabricated and finished at the same place, where the amount of oxidation formed on the metal surface for few days, will not contaminate **Picklex®**. **Note**: Depending on the condition of the metal parts, lot of time the pump and filtering system may not be needed for the dip tank with **Picklex®**.
- ♦ All dip tanks are made out of high density plastic only, reducing the set up cost considerably (old system had to use some metal tanks). The total payback time for setting up new process was 6 weeks (this includes the chemical cost to fill up the tank).
- Works at room temperature.
- Less energy consumption, because no heating is required for Picklex®
- Comparing with their old system with more number of steps and with longer dipping time, the total process time is reduced more than 50%, due to less number of steps in the new process and short application time.
- ♦ The running cost of the process is reduced more than 50%, because of very high surface coverage for **Picklex®** and no other added expenses (heating, waste disposal, monitoring etc.).
- Negligible evaporation loss from **Picklex®** tank, because the chemical is not heated.
- Cut down maintenance considerably
- ♦ Less labor, because no monitoring of pH or the thickness of coating is needed (**Picklex®** maintains the pH automatically).
- ♦ Easy cleanup for any spillage, because **Picklex®** is water soluble.
- Obtaining higher quality finishing.
- ♦ Later on, if this company decides to add different finishing lines, such as e-coating, anodizing or electroplating, they do not have to install a separate process for surface preparation/pre-treatment. The existing four step process can be used for that purpose, using the same grade of Picklex®.

P2 Technology Performance

This section will present the performance data for the P2 technology, as well as the quality performance of the finishing system due to this new P2 process by **Picklex®**, as a result of an actual application.

Also, this section will show the quality performance and P2 performance goal, which can be achieved in any other kind of finishing processes, by use of the same process used in this actual application.



P2 goals and objective achieved: P2 goals and objective were totally achieved by eliminating the hazardous waste disposal completely from the surface preparation/pre-treatment process. Also, there is **Zero Waste Discharge** from **Picklex®**. The rinse water is disposed after very long intervals, due to the face that, the reason for rinsing is just to remove the excess **Picklex®** from the surface of the metal parts. Unless the rinse water reaches a very high concentration of **Picklex®** (which will take a long period of time, because of the low viscosity of the chemical) and start dragging a considerable amount of chemical in the paint tank, it is not needed to be disposed. In case of powder coating, the rinsing is done to avoid the dust particles in the air inside the convection oven, to be attracted to the metal surface and contaminate the surface (the excess chemical makes a tacky surface in the beginning stage of drying). Rinse water can be disposed after neutralizing with caustic soda. Also, P2 goals were achieved for the chemical fumes (eliminated all hazardous fumes). Since **Picklex®** works at room temperature, no strong chemical fumes are present, which made the working conditions much better than before.

Due to a very large coverage of **Picklex®**, and very low evaporation loss, the running cost is reduced considerably. Also, due to less number of steps for the process, the process time is also reduced.

Note: Unlike acid wash, **Picklex® does not etch the metal surface**. It makes a metallurgical coating by chemically reacting with the metal surface and micro-rust (the coating becomes a part of the metal surface, less than ½ mil thick). Therefore no heavy metal goes to the rinse tank.

Summarizing the P2 goals and the performance:

- The hazardous waste was eliminated completely from surface preparation and pre-treatment process.
- Since no separate acid recovery system is needed in this new P2 technology using **Picklex®**, the total cost of having a recovery system is eliminated completely.
- ◆ The frequency of disposal for the rinse water after application of Picklex®, is very long, reducing labor and cost.
- ♦ Total energy cost for heating chemical and the maintenance are reduced considerably, since **Picklex®** works at room temperature.
- ◆ The overall efficiency of the finishing process is increased considerably.
- Reduced process time by more than 50%.
- Reduced running cost by more than 50%.
- Minimized final product rejection rate.
- Eliminated hazardous chemical fumes completely.
- Better working conditions

Cost Information

This section will present the cost information associated with **new installation** of this **alternate process** using **Picklex®** and also demonstrate how to use the same alternate process, replacing an **existing traditional process** of surface preparation and pre-treatment, using the existing equipment.



The cost associated with installing new surface preparation & pre-treatment process :

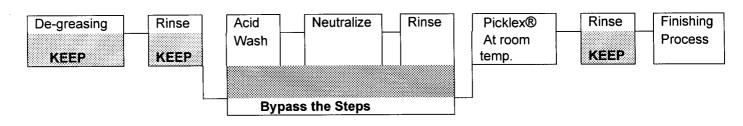
No metal tanks are required in this standard four step process. **Picklex®** can be used in a high density plastic or fiberglass tank. All four steps can be performed in plastic tanks, which reduces the equipment cost down to a minimum (in traditional process, metal tanks are used, some are stainless steel). Also, no heating system is required for **Picklex®**. Lot of time, depending on the condition of the metal parts, the pump and filtering system for **Picklex®** tank may not be necessary. The cost recovery time (which includes the cost of **Picklex®**) for this process, is anywhere from 2 to 4 months (the host plant in Texas recovered the cost in 6 weeks time). If compared with a seven stage surface preparation system with metal tanks, this four stage system along with the price of the chemicals to fill up the tanks, will be a fraction of the total cost to set up a traditional system. When in operation, since **Picklex®** has Zero Waste Discharge, there is no expense involved for waste disposal or setting up a recovery system for **Picklex®**.

The cost associated with switching a traditional process to the Standard Four Step Alternate Process with Picklex®™:

An example of a typical seven stage surface preparation & pre-treatment process presently used for metal finishing process is shown below.



Keeping all the equipment in same order, remove the contents from Acid tank through Phosphate tank, any one of those tanks can be used for **PICKLEX®**, so far it is made out of plastic or stainless steel. Keep the Degreasing, rinse and the final rinse tank can be used as is, as shown below.



Only cost associated with this change is to clean four tanks and fill up one tank with **Picklex®**. The cost recovery time should be within few weeks. After that time, the running cost will reduce considerably, but process time will remain the same (unless three tanks are removed from the middle and the process is revised to a standard four-step process, as shown before).



The operating cost for this alternate process is around 50% lower than the traditional process (this is an average figure estimated, from the actual percentage reduction in cost occurred in the commercial operation in Texas), due the following facts:

- > Zero Waste Discharge for Picklex® cuts down waste disposal time, labor and cost.
- > Negligible evaporation loss, because **Picklex**® works at room temperature.
- Less equipment to maintain, due to fewer steps and no heating for **Picklex**® tank.
- > No monitoring of pH and coating thickness cuts down labor and cost.
- The same standard four-step process by **Picklex**® works on steel and aluminum, and also for different finishing systems. For example, a single four-step process can be used for both Electroplating as well as Anodizing at the same time. This eliminates a complete surface preparation process, reducing a set up cost and also the operating cost.

Regulatory/Safety Requirements

For using **Picklex**® and the rinse water afterwards, in the surface preparation process, there is no known regulatory requirements. When the rinse water with some **Picklex**® is ready to be disposed, it has to be neutralized with caustic soda. This alternate process does not fall under any formal EPA or OSHA regulation (also, there is no D. O. T. regulation requirement while shipping).

Health/Safety Issues	
Health/Safety issues	

For this alternate process, there is no significant health hazard present, because there is no hazardous/toxic chemicals used. Also, the only tank which is heated is the de-greasing tank, rest of the tanks operates at room temperature. **Picklex**® is very safe to use and easy to clean, if spilled. Also, it is non-toxic, non-hazardous, non-flammable and water soluble. Very less training is necessary for operator safety.

Lessons Learned / Implementation Issues

Lessons Learned

The following lessons were learned during the development of the technology, and during commercial use:

- > This alternate process eliminates hazardous waste from metal surface preparation and pre-treatment process.
- Eliminates hazardous and toxic chemicals from surface preparation/pre-treatment process before any metal finishing.
- > Replaces the phosphate line completely, i.e., **Picklex**® replaces acid wash through phosphate treatment. **Picklex**® can replace the passivation of steel, which is done by acid.
- > Eliminates all pollution (ground and air) presently created by the hazardous chemicals, such as acid, phosphate etc.
- > Because Picklex® works at room temperature, the chemical fumes are cut down to a minimum.
- > Eliminates EPA and OSHA problems.
- > Simplifies the metal surface preparation process considerably.
- > Eliminate Flash Rust problem
- > Produces much superior quality finishing.
- > The Picklex® coating stops creeping of rust later on.
- Same grade of Picklex® works for both steel and aluminum.
- > All metal finishing systems can use the same standard four step surface preparation/pre-treatment process with **Picklex®**
- > Overall process time, labor and running cost reduces considerably, due to less number of stages, less monitoring (no monitoring of pH or the thickness of the coating is needed) and very high coverage of **Picklex®**
- When water is dragged from the rinse water tank to the **Picklex®** tank, it should be within 15%, otherwise the quality of the coating may be affected.
- > Picklex® should be kept reasonably clean by proper filtering system.
- Except steel and aluminum, no other metal has been tested yet using Picklex®.

Implementation Considerations

Based on the experience with this technology application and also from all the field tests, including the laboratory tests on different kinds of metal finishing processes, lesson learned relative to the future implementation of this technology with the product **Picklex®** are shown below. The quality performances noticed during laboratory tests and field tests are also described.

Quality Performance & P2 Performance Goal for Other Finishing Systems:

Along with Powder coating and Liquid Painting, the alternate process for surface preparation & pre-treatment can be used in the following finishing systems also:

- Electroplating
- Anodizing
- E-coating
- Metal Pickling Process (De-scaling/Pickling)

All the above finishing systems (except metal pickling), can also use the same four step standard process with **Picklex®**, to achieve the same P2 performance goals as achieved by the host plant in their painting and powder coating processes. This P2 technology with the product **Picklex®**, can also be applied during de-scaling and pickling of metal after heat treatment and annealing process. In one step dipping process, the **Picklex®** will descale, condition the metal and provide a metallurgical protective coating, all at the same time with Zero Waste Discharge, replacing the conventional pickling process with acid & phosphate completely.

Note: This process does not cause any Hydrogen embrittlement during de-scaling/pickling.

Along with solving the pollution problem and reducing process time & cost in the metal surface preparation area, this alternate process also provides quality results for all metal finishing processes. The laboratory test results and field test results are summarized below:

Electroplating:

Field tested by **US EPA** (evaluation under Zero Waste Discharge program), where it passed all required quality tests. The surface preparation was done by standard four steps, de-greasing, rinse, dipping in **Picklex®** at room temperature and rinsed before electroplating. Passed all quality tests.

Anodizing:

Tested for more than **500 hrs** (MIL-C-5541 and ASTM B-117). The surface preparation and pre-treatment were done, by just dipping the aluminum panels in **Picklex®**, and a quick rinse before anodizing. Also field tested by **US EPA** (evaluation under Zero Waste Discharge program), where anodizing was done after two step surface preparation by **Picklex®** at room temperature, and passed all required quality tests including **500 hours** of salt spray test (continuing).

Powder Coating:

Field tested by Hughes Parker Co. on steel and passed all quality tests including **1500 hours** of salt spray test (ASTM B117).

Field tested by Custom Finishing Co., Birmingham, AL for Powder Coating on aluminum, after treating with **Picklex®** at room temperature. Adhesion, bending and impact tests passed. Also, it passed **500 hours** of salt spray test.

Painting:

Picklex[™] has been tested for **1540 hours** of salt spray (ASTM B-117) with minimum surface preparation. Hot rolled steel test panels with mill scale were cleaned and coated by just dipping in **Picklex®**, before painting process.

E-Coat:

Field tested by Hughes Parker Co., Lawrenceburg, TN. The surface preparation was done by standard four step process. Passed all quality tests and **1500 hours** of salt spray (ASTM B117).

Conclusion

The **Alternate Standard Four Step Process**, with the unique product **Picklex®**, not only solves the waste and pollution problems, but also it is very simple & safe to use. Also, it gives the metal finishers a chance to provide the customers with a higher quality finishing.

ATTACHMENT:

- 1) <u>Letters from US EPA</u>
- 2) <u>Lab Test on Painting</u>
- 3) <u>Lab Test on Anodizing</u>



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICE OF RESEARCH AND DEVELOPMENT NATIONAL RISK MANAGEMENT RESEARCH LABORATORY CINCINNATI, OHIO 45268

Ranjit Sen
International Chemical Products, Inc.
1209 Meadow Park drive
Huntsville, AL 35803

Subject: Zero Waste Discharge during metal surface preparation process

Dear Mr Sen,

After your presentation in Cleveland, OH last month, we are interested to perform further evaluation and testing of the product PicklexTM, as a possible replacement of the hazardous and/or toxic chemicals, presently used for many surface preparation and pre-treatment processes before metal finishing (with no waste disposal).

We already conducted the first test using PicklexTM as a pretreatment to Hard Chromium electroplating on Sept10th 1998. Eight steel test panels (four panels with visible surface rust & oil, and the other four panels without visible rust and oil) were used for the test. Surface preparation & pre-treatment was conducted as follows:

- 1. The test panels were dipped in a de-greasing solution
- 2. Then rinsed in a rinse tank
- 3. Dipped in PicklexTM at room temperature for 10 minutes
- 4. Rinsed in DI water
- 5. Hard Chrome plating for 3 hours at 3 amps/sq.in.

The test panels showed no visual signs of quality problems, however we did perform some quality testing as follows:

Adhesion testing: Bending test was performed on four panels, and passed. There was no flaking or peeling of the coating.(ASTM B571-91)

Porosity test: Porosity test was performed on one panel, which had surface rust before pretreatment, and also on another panel which had no visible rust before pretreatment with PicklexTM. (Ferroxyl Test- This method reveals discontinuities, such as pores or pits in coatings of chromium.) Both panels passed the test.

The process and the test results were quite impressive and now we are in the process of putting together a workplan for peer review scrutiny and funding approval for testing the product as a pretreatment for different kinds of metal finishing, such as, anodizing, powder coating, e-coat, pickling (with evaluation of the hydrogen embrittlement) etc.

Please provide me with the detail surface preparation process/procedures with PicklexTM, for all the above metal finishing processes. Also, identify the ASTM tests required for those processes to your knowledge.

Sincerely

T. David Ferguson



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICE OF RESEARCH AND DEVELOPMENT NATIONAL RISK MANAGEMENT RESEARCH LABORATORY CINCINNATI, OHIO 45268

December 1,1998

Ranjit Sen
International Chemical Products, Inc.
1209 Meadow Park drive
Huntsville, AL 35803

Subject: Testing of PicklexTM

Dear Mr Sen,

As you are aware, we have already conducted the first test using PicklexTM as a pretreatment to Hard Chromium electroplating on Sept10th 1998. The test panels showed no visual signs of quality problems, and adhesion testing and porosity testing was performed on the panels.

The process and the test results were quite impressive and so we tried the pretreatment for anodizing. On five aluminum test panels we performed the following procedure.

- 1. 10 minutes in PicklexTM
- 2. Quick rinse
- 3. 35 minutes sulfuric anodize (Type II, Class I)
- 4. Rinse
- 5. 25 minute hot water seal

The panels passed the 500 hour salt spray test and we are planning to put the panels back into the salt spray chamber until failure. We also tested the panels with the gold chemical film anodizing, however those panels did not pass the salt spay test.

The results of the Sulfuric Anodizing (Type II, Class I) are very impressive and we would like to perform more research on PicklexTM in the future. These are preliminary tests and more in depth testing is needed. I look forward to working with you and your product in the future.

Sincerely

Γ. David Ferguson



ENVIRONMENTAL TEST REPORT

Submitted for testing by:

International Chemical Products, Inc. P.O. Box 4246 Huntsville, AL 35815-4246

Prepared by:

TMC, Inc. 210 Wynn Drive Huntsville, AL 35805

June 03, 1997

Test Report No. 37101-1

Analysis performed by: Alex Gallien

Approved for distribution:

Bharti Ujjani, Vice President Lab Services

INTRODUCTION:

ICP, Inc. requested analysis of five (5) hot rolled sheet metal panels on 03-28-97. The analysis consisted of salt spray testing as per the Test Method MIL-C-5541, para 4.5.1 and ASTM B117. The surface preparation of all the panels was performed by ICP personnel according to the attached procedure.

SAMPLE INFORMATION:

A total of five (5) panels were submitted for salt spray testing. Panels submitted were as follows:

Panel Type	Quantity	<u>Description</u>
Hot Rolled Sheet Metal Panels	5	Mill Scale and Surface rust removed with PICKLEX TM as described in the attached procedure

SAMPLE PREPARATION:

Sample test panels were treated as described in Attachment I prior to Salt Spray Test. The sample size were of 3" x 8" in dimensions.

SALT SPRAY TEST METHOD & EVALUATION:

Salt spray test was performed as per the test method MIL-C-5541 and ASTM Bll7. The panels shall show no more than 5 pits, none larger than 1/32" in diameter in a 30 square inch area, with the exception of 1/4" around the edge.

RESULTS:

The panels were placed in the chamber at 10:00 a.m. on 03-31-97. They were removed and inspected at 10:00 a.m. 04-22-97 after 528 hours of exposure. The panels did not exhibit any signs of corrosion including pitting or blisters. The panels were placed back into the chamber and further exposed to salt fog for an additional 1012 hours for a total of 1540 hours and removed at 3:00 p.m. on 6-3-97. At the end of 1540 hours, the panels did not exhibit any pits; however there was blistering of paint originating at the edges of the panels.

This is to certify that the five (5) hot rolled sheet metal panels do meet the requirements of MIL-C-5541 and ASTM Bll7 salt spray test.

Preparation of the test panels for salt spray test:

A set of 5 hot rolled sheet metal panels were submitted to TMC laboratory by International Chemical Products, Inc., to conduct a salt fog test, and according to ICP, Inc., the following procedure was followed for surface preparation of the sheet metal panels and painting.

Note: The surface preparation of the sheet metal panels were done by the a specific method, totally different from the standard industrial method for surface preparation (pickling/cleaning) of sheet metal.

Product used: PICKLEXTM 20

Step 1: A set of five hot rolled sheet metal panels were obtained from a sheet metal shop. The panels had dark mill scale, mill oil, some surface rust and dust. The edges of the panels were unfinished.

Step 2: The panels were dipped in PICKLEXTM for around 35 minutes at ambient temperature. By that time all the mill scale and surface rust came loose and were taken off the surface by light brushing by a paint brush (the mill oil and dust were removed by PICKLEXTM within one minute from dipping). There was no mill scale or surface rust left on the metal surface.

Note: This one step dipping process to completely remove mill scale, mill oil, surface rust and dust from the sheet metal surface, was witnessed by TMC laboratory personnel. Then the panels were left for drying. After drying, light shades of gray color were noticed on the surface of the panels, which was due to the complete conversion of the micro-rust on the metal surface to a protective coating.

Step 3: The panels were painted according to the following procedure:

- a) The surface was lightly sanded with 0320 sand paper.
- b) Paint sealer was applied on the surface with Dupont Nason paint sealer.
- c) Surface painted with Dupont Nason Automotive Polyurethene Paint (white).
- d) Dried for 24 hours.

Step 5: Submitted to TMC laboratory for testing.



ENVIRONMENTAL TEST REPORT

INTERNATIONAL CHEMICAL PRODUCTS, INC. P.O. Box 4246 Huntsville, Alabama 35815-4246

Prepared by:

TMC, INC. 210 Wynn Drive Huntsville, Alabama 35805

February 26, 1998

Test Report No. 28012-2

Analysis performed by: A. Gallien

Approved for distribution:

Bharti Ujjani, Vice President Lab Services

INTRODUCTION

International Chemical Products requested analysis of five (5) aluminum alloy panels on 02-04-98. The analysis consisted of Salt Spray Test Method **ASTM B117** and **MIL-C-5541** (186 hrs.). The samples were identified as follows:

Quantity

Description

5

Aluminum Test Panels

SURFACE PREPARATION AND ANODIZING INFORMATION: Surface preparation of the test panels and anodizing were performed by J & A Finishing Co., Huntsville, Alabama as follows:

Panels were dipped in solution provided by the customer (PicklexTM) for 2 minutes at 130°F. The parts were rinsed in tap water, then put in Anodize solution for 25 minutes at 16 volts. The Mil-Spec for Anodize is MIL-A-8625, Type II, Class 1.

ENVIRONMENTAL TESTS AND EVALUATION

Salt Spray Test Methods ASTM B117 and MIL-C-5541: The panels shall show no more than 5 pits none larger than 1/32" in diameter in a 30 square inch area excepting 1/4" around the edge.

RESULTS

The panels were placed in the salt chamber at 4:30 p.m. on 02-04-98 and removed at 4:30 p.m. on 02-25-98. All panels exhibited no evidence of corrosion products after 504 hours, therefore meeting the criteria for passing.

This is certify that the five (5) aluminum alloy panels meet the requirements of MIL-C-5541 and ATM B117.